

**REMARKS**

Claims 1-5 are pending.

**I. Amendments**

In order to more clearly define the present invention, Applicants have instantly amended the present specification and claims. The gist of the amendments is as follows.

**(I-1) Amendments to the specification**

Amendment to page 23, line 10 is merely a correction of an inadvertent error which occurred at the time of the translation into English of the original Japanese PCT specification. Specifically, the number “0.06” has instantly been corrected to —0.60--.

Amendment to page 66, line 2 is also a correction of an inadvertent error which occurred at the time of the translation into English of the original Japanese PCT specification. Specifically, the number “5” has instantly been corrected to--0.5--.

In support of the above-mentioned amendments to page 23, line 10 and page 66, line 2, the Applicant attaches hereto a DECLARATION to verify that the amendments are made only to correct inadvertent errors which occurred at the time of the translation into English of the original Japanese PCT specification.

Amendments to page 59, line 1, page 75, lines 4, 6 and 9, page 85, line 24, page 95, line 17, page 99, line 19 and Table 1 at page 111 are merely corrections of clerical errors.

**(I-2) Amendments to the claims**

In claim 1, “separating” in step (3) has been amended to “recovering”. Support for this amendment is found at, for example, page 83, line 17 of the present specification.

No new matter has been added by way of the above-amendment.

## **II. Information Disclosure Statement**

In the outstanding office action, the Examiner states that the information disclosure statement filed on July 7, 2004 fails to comply with 37 CFR 1.98(a)(2). Specifically, the Examiner states that the copies of the Japanese references are missing from the PTO's electronic copy of the captioned application.

### **Applicants respond as follows.**

Applicants note that this application is a National Phase of a PCT application and the Japanese references were cited on the International Search Report. Accordingly, copies of the references should have been forwarded from the International Search Authority. However, in order to advance prosecution, Applicants enclose herewith copies of JP 9-2998, JP 62-77349, JP 5-92102 and JP 8-268955 with English abstracts thereof for the Examiner's consideration. In addition, Applicants enclose herewith a new PTO-1449 form for the Examiner's signature.

Thus, it is believed that the objection with respect to information disclosure statement should be withdrawn.

## **III. The state of the art and the features and advantages of the present invention**

Before specifically addressing the rejection of the claims over the cited document, it is believed that the following background information should be considered in order to shed a proper light on the development of the present invention and the advantageous features thereof.

As discussed in the specification under "Prior Art", conventionally, glycolic acid has been used as an important component of cosmetics, a hair dying agent, a shampoo, detergents (e.g., a detergent for domestic use and a detergent for industrial use), a metal treating agent, a tanning agent and the like. In recent years, glycolic acid is also widely used as a raw material for various chemical products, a raw material for various synthetic resins and the like.

It is required that glycolic acid for use as the above-mentioned raw materials have high purity (**as high as 99.9 % or more**). The reason for this is explained below, taking as an example the case where glycolic acid is used as a raw material for a synthetic resin, wherein the glycolic acid is required to have an extremely high purity.

In general, for obtaining a polyester resin comprised mainly of glycolic acid monomer units, which exhibits a mechanical strength sufficient for general use, it is desired that the polyester resin have a weight average molecular weight of 150,000 or more. When such a polyester resin is produced by polycondensation of a raw material mixture containing glycolic acid as a main component thereof, care must be taken as to the following points. Glycolic acid is a self-condensing compound having, in a molecule thereof, one carboxyl group and one alcoholic hydroxyl group. Therefore, when the raw material mixture contains, as an impurity, a carboxylic acid having no alcoholic hydroxyl group or an alcohol having no carboxyl group, such an impurity inevitably reacts with glycolic acid and/or a polycondensation product of glycolic acid, thereby terminating the growth of the polymer chain. As a result, a polyester resin having a high molecular weight cannot be obtained. Further, when the raw material mixture contains a salt as an impurity, the mechanical property of the polyester resin obtained is markedly lowered. Accordingly, when the production of a polyester resin is performed by polycondensation of a raw material mixture containing glycolic acid as a main component thereof, it is necessary that the glycolic acid have a high purity. In general, the glycolic acid is required to have **a purity as high as 99.9 % by weight or more**.

As conventional methods for producing glycolic acid on a commercial scale, there can be mentioned:

- a) a method in which glycolic acid is produced by reacting formaldehyde, carbon monoxide and water in the presence of an acid catalyst; and
- (b) a method in which chloroacetic acid is saponified.

In each of the above-mentioned methods (a) and (b), glycolic acid is obtained in the form of an aqueous solution thereof. However, in each of these methods, a carboxylic acid and an alcohol are **by-produced in large amounts**, and a salt (**which is an impurity**) is by-produced in an amount such that the mechanical properties of a resin produced from the glycolic acid become markedly low. Therefore, for obtaining a high purity glycolic acid which can be used as a raw material for producing a resin having a high molecular weight, it is necessary that the aqueous glycolic acid solution be subjected to purification.

As an example of a generally employed purification method, there can be mentioned a distillation method. When the purification of glycolic acid is performed by distillation method, a distillate containing glycolic acid is obtained, and the obtained distillate is cooled and solidified to thereby obtain high purity glycolic acid crystals. However, it is difficult to obtain high purity glycolic acid crystals by the distillation of the above-mentioned aqueous solution of glycolic acid. The reason for this is as follows. Glycolic acid has low volatility. Further, glycolic acid is susceptible to a polycondensation reaction under the distillation conditions, so that a condensation product which is difficult to distill is formed. Therefore, it is difficult to obtain high purity glycolic acid crystals by the distillation method.

For these reasons, there have been proposed methods (other than the distillation method) for producing high purity glycolic acid crystals from an aqueous glycolic acid solution.

For example, as a method for producing glycolic acid crystals from an aqueous glycolic acid solution, there can be mentioned Japanese Patent Application Prior-to-Examination Publication (Tokuhyo) No. Hei 6-501268 (**corresponding to WO92/05138 cited in the outstanding office action**) describes a method in which an aqueous glycolic acid solution containing about 70 % by weight of monomeric glycolic acid is subjected to a specific crystallization treatment to thereby obtain glycolic acid crystals. In this patent document, it is described that high purity glycolic acid crystals can be obtained depending on the conditions for crystallization of glycolic acid. However, when it is intended to obtain high purity glycolic acid crystals in high yield by the method described in the patent document, a very long period of time

is needed for crystallization of glycolic acid and, thus, the method becomes economically disadvantageous.

Thus, by the conventional methods, it has been impossible to produce high purity glycolic acid crystals from an aqueous glycolic acid solution easily and in high yield on a commercial scale.

In this situation, the present inventors have made extensive and intensive studies with a view toward developing a method for producing high purity glycolic acid crystals (i.e., glycolic acid crystals having **a purity of 99.9 % or more**) from an aqueous glycolic acid solution easily and in high yield on a commercial scale. As a result, it has unexpectedly been found that, by subjecting a specific aqueous glycolic acid solution to crystallization, it becomes possible to produce high purity glycolic acid crystals (i.e., glycolic acid crystals having **a purity of 99.9 % or more**) **easily and in high yield** on a commercial scale, which crystals are substantially free of impurities, such as carboxylic acids other than glycolic acid, alcohols and salts. The specific aqueous glycolic acid solution contains monomeric glycolic acid and a glycolic acid condensation product, and has **a specific water content, a specific calculated monomeric glycolic acid weight ratio** (ratio of the total weight of the monomeric glycolic acid and the component monomeric glycolic acid of the glycolic acid condensation product to the weight of the aqueous solution) and **a specific monomeric glycolic acid content**. Based on this novel finding, the present invention has been completed.

Specifically, the amended claim 1 of the present application (hereinafter, simply referred to as "claim 1 of the present application") is as follows.

1. A method for producing high purity glycolic acid crystals from an aqueous glycolic acid solution, which comprises the steps of:

(1) providing an aqueous glycolic acid solution (A) containing monomeric glycolic acid and a glycolic acid condensation product, said aqueous glycolic acid solution (A) having the following characteristics (a), (b) and (c):

- (a) a water content of from 5 to 20 % by weight,
- (b) a calculated monomeric glycolic acid weight ratio of from 0.60 to

1.00, said calculated monomeric glycolic acid weight ratio being defined as a ratio of the total weight of said monomeric glycolic acid and said glycolic acid condensation product to the weight of said aqueous solution (A), wherein the weight of said glycolic acid condensation product is expressed in terms of the weight of component monomeric glycolic acid of the glycolic acid condensation product, and

- (c) a monomeric glycolic acid content of from 20 to 57 % by weight,
- (2) depositing glycolic acid crystals from said aqueous glycolic acid solution (A), and
- (3) recovering the deposited glycolic acid crystals from said aqueous glycolic acid solution (A).

As is apparent from claim 1 above, the **most important feature** of the method of the present invention is the use of aqueous glycolic acid solution (A) having the above-mentioned **characteristics (a)** (a water content of from 5 to 20 % by weight), **(b)** (a calculated monomeric glycolic acid weight ratio of from 0.60 to 1.00) and **(c)** (a monomeric glycolic acid content of from 20 to 57 % by weight). By using the above-mentioned aqueous glycolic acid solution (A), it has, for the first time, become possible to produce **high purity** glycolic acid crystals easily and in **high yield** within a **short crystallization time**.

For achieving the **excellent effects of the present invention** (i.e., producing **high purity** glycolic acid crystals easily and in **high yield** within a **short crystallization time**), it is **critical** that aqueous glycolic acid solution (A) have the above-mentioned characteristics (a), (b) and (c). In order to substantiate this contention of the Applicants, the Applicants have made observations, with reference to Examples 1 to 5 and Comparative Examples 1 to 6 of the present application, to show that, the above-mentioned characteristics (a), (b) and (c) of the aqueous glycolic acid solution (A) are critical for achieving the excellent effects of the present invention (i.e., producing **high purity** glycolic acid crystals easily and in **high yield** within a **short crystallization time**). The method and results are as described in Exhibit 1 of the accompanying executed Mr. Kuroda Declaration.

For easy reference, Table A (summarizing the results of Examples 1 to 5 and Comparative Examples 1 to 6) shown in Exhibit 1 is reproduced below.

Table A

	Glycolic acid solution used in deposition step			Deposition step	Glycolic acid crystals produced	
	Water content (wt%) (Characteristic (a): 5-20 wt%)	calculated monomeric glycolic acid weight ratio (Characteristic (b): 0.6-1)	monomeric glycolic acid content (wt%) (Characteristic (c): 20-57]	Crystallization time (hours)	Yield (wt%)	Purity (wt%)
Ex. 1	18	0.83	54.5	0.25	27	99.96
Ex. 2	18	0.83	54.5	0.25	20	99.96
Ex. 3	13	0.91	52	2	28	99.97
Ex. 4	13	0.91	52	2	28	99.92
Ex. 5	7	1.00	41	0.75	23	99.90
Comp. Ex. 1	31 (*1)	0.70	59 (*3)	1.5	20	99.47
Comp. Ex. 2	31 (*1)	0.70	59 (*3)	1.5	20	99.55
Comp. Ex. 3	3 (*1)	1.05 (*2)	35	1	20	99.68
Comp. Ex. 4	19	0.84	65 (*3)	0.75	23	99.69
Comp. Ex. 5	23 (*1)	0.75	55	0.75	14	99.72
Comp. Ex. 6	6	1.02 (*2)	34	0.75	22	99.70

Notes: (\*1) outside the range of characteristic (a)

(\*2) outside the range of characteristic (b)

(\*3) outside the range of characteristic (c)

It can be fairly concluded from the results:

(1) that, in each of Examples 1 to 5 in which aqueous glycolic acid solutions satisfying the above-mentioned characteristics (a), (b) and (c) were used, there were produced glycolic acid crystals having a purity as high as from 99.90 to 99.97 % in a yield of as high as from 20 to 27 % by weight within a crystallization time as short as from 0.25 to 2 hours;

(2) that, on the other hand, in Comparative Examples 1 to 6 in which aqueous glycolic acid solutions which do not satisfy at least one of the above-mentioned characteristics (a) (b) and (c) were used, there were produced glycolic acid crystals having a **purity of only from 99.47 to 99.72 % (i.e., lower than 99.90 %, which is aimed at by the present invention)**; and

(3) that, from items (1) and (2) above, it is apparent that the above-mentioned characteristics (a), (b) and (c) of aqueous glycolic acid solution (A) are **critical** for achieving the excellent effects of the present invention (i.e., producing **high purity** glycolic acid crystals easily and in **high yield** within a **short crystallization time**).

#### IV. Rejection of the claims under 35 U.S.C. §112

Turning now to the rejection of the claims, claims 1 to 5 have been rejected under 35 U.S.C. §112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

Specifically, the examiner states that it is unclear how the term “depositing” in step (2) of claim 1 is different from the term “separating” in step (3) of claim 1, since the broadest reasonable interpretation of “separating” can necessarily include “depositing”.

**Applicants respectfully traverse the rejection.**

As mentioned in item (II-2) above, Applicants have replaced the term “separating” in step (3) with “recovering”. By this amendment, it is apparent that the method of the present invention comprises (1) providing a specific aqueous glycolic acid solution (A), (2) depositing glycolic acid crystals from aqueous glycolic acid solution (A), and (3) recovering the deposited glycolic acid crystals from aqueous glycolic acid solution (A).

Thus, it is believed that the rejection of claims 1 to 5 under 35 U.S.C. 112 should be withdrawn.

## V. Rejection of the claims under 35 U.S.C. 102(b)

Further, claims 1 to 3 have been rejected under 35 U.S.C. 102(b) as being anticipated by WO92/05138 (**WO 138**). Specifically, the Examiner states that **WO 138** teaches a glycolic acid product of a carbonylation reaction given as the tables on pages 3 and 4, and that a seeded crystallization of glycolic acid crystals in the glycolic acid solution is given on pages 5 and 6, at a preferred temperature of 5 °C.

**Traverse is respectfully made as follows.**

**WO 138** teaches a method for producing high-purity, crystalline hydroxyacetic acid (i.e., glycolic acid). However, **WO 138** has no teaching or suggestion about the criticality of the above-mentioned characteristics (a) (a water content of from 5 to 20 % by weight), (b) (a calculated monomeric glycolic acid weight ratio of from 0.60 to 1.00) and (c) (a monomeric glycolic acid content of from 20 to 57 % by weight) which are defined in claim 1 of the present application for simultaneously achieving **high purity, high yield and short crystallization time**. More specific explanation is given below.

In **WO 138**, the aqueous glycolic acid solution used is a 70 % technical grade aqueous solution (see page 3, lines 14 to 15 of **WO 138**). That is, the glycolic acid solution produced in **WO 138** has a water content of 30 % by weight, which is outside the range of characteristic (a) (5 to 20 % by weight) defined in claim 1 of the present application. Therefore, in **WO 138**, the excellent effects of the present invention (**high purity, high yield and short crystallization time**) are not achieved. In order to substantiate this contention of the Applicants, the Applicants have made observations, with reference to Examples 1 to 5 of **WO 138** and working examples of the present application, to show that the excellent effects of the present invention (i.e., **high purity, high yield and short crystallization time**) are not achieved in **WO 138** (in which characteristic (a) defined in claim 1 of the present invention is not satisfied). The method and results are as described in Exhibit 2 of the accompanying Declaration under 37 CFR 1.132 by one of the coinventors, Mr. Yoshito Kuroda.

For easy reference, the results of the working examples of **WO 138** and the present invention are summarized below in Table B.

Table B

		Purity (wt %)	Yield (wt %)	Crystallization time (Hours)
Working examples of <b>WO 138</b>	Ex. 1	99.9	<b>6.6</b>	24
	Ex. 2	99.4	23.6	0.25
	Ex. 3	99.3	16.7	5
	Ex. 4	99.9	27	<b>63</b>
	Ex. 5	99.8	19.0	65
Working examples of the <b>present application</b>		99.90 to 99.97	20 to 27	0.25 to 2

It can be fairly concluded from the results:

- (1) that, in Examples 2, 3 and 5 of **WO 138**, the purity is as low as from 99.3 to 99.8 (vs. 99.90 % by weight, which is aimed at by the present invention);
- (2) that, in Example 1 of **WO 138**, although the purity is 99.9 %, the **yield** is as low as 6.6 % (vs. 20 % by weight, which is achieved in the working examples of the present application);
- (3) that, in Example 4 of **WO 138**, although the purity is 99.9 %, the **crystallization time** is disadvantageously long as **63 hours** (vs. 0.25 to 2 hours, which is achieved in the present invention); and
- (4) that, from items (1) and (3) above, it is apparent that the excellent effects of the present invention (i.e., **high purity, high yield and short crystallization time**) are not achieved in **WO 138**.

Further, to more clearly substantiate the inventive step of the present invention over **WO 138**, the Applicant has made observations to show that the method of **WO 138** is

**disadvantageous** as compared to the method of the present invention, with reference to Examples 1 and 4 of **WO 138** and working examples of the present application. The method and results are as described in Exhibit 3 of the accompanying executed Mr. Kuroda Declaration.

As observed in Exhibit 3 of the accompanying Mr. Kuroda Declaration, attention is drawn to Examples 1 and 4 of **WO 138**. In Example 1 of **WO 138**, although the purity is as high as 99.9 % (which is aimed at by the present invention), the **yield** is as low as **6.6 % by weight** (vs. **20 % by weight** achieved in the working examples of the present application). On the other hand, in Example 4 of **WO 138**, although the purity is as high as 99.9 %, the crystallization time is as long as **63 hours** (vs. **0.25 to 2 hours** achieved in the working examples of the present application).

With respect to the disadvantages of such a low yield and a long crystallization time, the following should be noted.

(1) In the case where the yield is as low as **6.6 % by weight** (i.e., Example 1 of **WO 138**), when it is intended to produce a predetermined amount of glycolic acid crystals within a predetermined crystallization time, as can be seen from formula (i) below, the amount of aqueous glycolic acid solution needed for the production will be **3 times** the amount of aqueous glycolic acid solution needed in the case where the yield is at least **20 % by weight** (i.e., present invention):

$$20/6.6 = 3 \quad (i)$$

When the amount of glycolic acid solution needed becomes 3 times larger, the **energy** needed for adjusting temperature (i.e., cooling) during the crystallization step would be **at least 3 times larger** than the energy needed in the present invention. Further, when the amount of glycolic acid solution needed becomes 3 times larger, it becomes necessary to **scale-up the production system**. Needless to say, in the production of glycolic acid crystals on a commercial scale (e.g., plant production), in such a case where the consumed energy becomes 3 times larger,

and there is a need to scale-up the production system, the **cost** would inevitably become disadvantageously **high**, as compared to the case of the present invention.

(2) In the case where the crystallization time is as long as **63 hours**, when it is intended to produce a predetermined amount of glycolic acid crystals within a predetermined crystallization time, as can be seen from formula (ii) below, the amount of aqueous glycolic acid solution needed for the production will be **31.5 times** the amount of aqueous glycolic acid solution needed in the present invention (in which the yield is not more than **2 hours**):

$$63/2 = \mathbf{31.5} \quad (\text{ii})$$

When the amount of glycolic acid solution needed becomes 31.5 times larger, the **energy** needed for adjusting temperature (i.e., cooling) during the crystallization step would be **at least 31.5 times larger** than the energy needed in the present invention. Further, when the amount of glycolic acid solution needed becomes 31.5 times larger, it becomes necessary to **scale-up the production system**. Needless to say, in the production of glycolic acid crystals on a commercial scale (e.g., plant production), in such a case where the consumed energy becomes 31.5 times larger, and there is a need to scale-up the production system, the **cost** would inevitably become disadvantageously **high**, as compared to the case of the present invention.

(3) Further, for reference, in the case where the yield is as low as **6.6 % by weight** and the crystallization time is as long as **63 hours**, when it is intended to produce a predetermined amount of glycolic acid crystals within a predetermined crystallization time, as can be seen from formula (i) below, the amount of aqueous glycolic acid solution needed for the production will be **at least 95.5 times larger** than the amount of aqueous glycolic acid solution needed in the present invention (in which the **yield is at least 20 % by weight and the crystallization time is not more than 2 hours**):

$$(20/6.6) \times (63/2) = \mathbf{95.5} \quad (\text{iii})$$

When the amount of glycolic acid solution needed becomes 95.5 times larger, the **energy** needed for adjusting the temperature (i.e., cooling) during the crystallization step would be **at least 95.5 times larger** than the energy needed in the present invention. Further, when the amount of glycolic acid solution needed becomes 95.5 times larger, it becomes necessary to **scale-up the production system significantly**. Needless to say, in the production of glycolic acid crystals on a commercial scale (e.g., plant production), in such a case where the consumed energy becomes 95.5 times larger, and there is a need to scale-up the production system significantly, the **cost** would inevitably become significantly **high**, as compared to the case of the present invention.

Therefore, it is apparent that, the method of **WO 138** is **disadvantageous** as compared to the method of the present invention.

From the above, it is apparent that claims 1 to 3 of the present application have patentability over **WO 138**.

## **VI. Rejection of the claims under 35 U.S.C. 103(a)**

Furthermore, claims 4 and 5 have been rejected under 35 U.S.C. 103(a) as being unpatentable over Applicant's admission of prior art in view of WO92/05138 (**WO 138**).

However, claims 4 and 5 depend from claim 1. Therefore, now that claim 1 has been substantiated to have a full patentability over **WO 138**, claims 4 and 5 have patentability over **WO 138**.

From the above, it is believed that the rejection to claims 4 and 5 under 35 U.S.C §103(a) should be withdrawn.

## VII. Conclusion

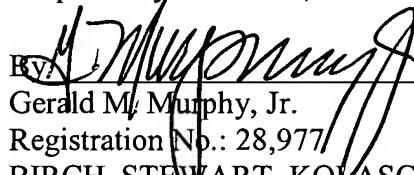
From the foregoing, it is firmly believed that all the rejections and objections should be withdrawn.

In view of the above amendment, applicant believes the pending application is in condition for allowance.

Should there be any outstanding matters that need to be resolved in the present application, the Examiner is respectfully requested to contact Garth M. Dahlen, Ph.D., Esq. (Reg. No. 43,575) at the telephone number of the undersigned below, to conduct an interview in an effort to expedite prosecution in connection with the present application.

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Respectfully submitted,

  
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Enclosures: 1) copies of JP 9-2998, JP 62-77349, JP 5-92102 and JP 8-268955 with English abstracts thereof,  
2) PTO-1449 form listing the JP references  
3) Declaration by Mr. Ryu Miyamoto regarding translation errors  
4) Rule 132 Declaration by Mr. Yoshito Kuroda